

Section 1.0  
Revision 0  
May 1991  
Page 1 of 57

DIOXINS ONLY



ANALYTICAL SERVICES  
QUALITY ASSURANCE PROJECT PLAN


FOR

RCRA FACILITY INVESTIGATION  
CIBA-GEIGY FACILITY  
CRANSTON, RHODE ISLAND

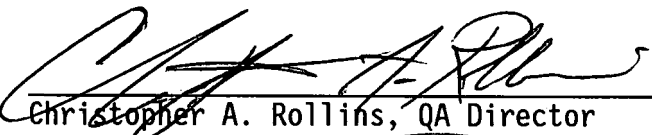
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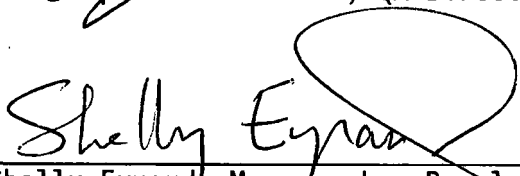
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Enseco-California Analytical Laboratory  
RCRA Facility Investigation  
Analytical Quality Assurance Project Plan  
Table of Contents

Section 1.0	Title Page
Section 2.0	Table of Contents
Section 3.0	Project Description
Section 4.0	Responsibilities and Authorities
Section 5.0	Quality Assurance Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness and Comparability
Section 6.0	Sampling Procedures
Section 7.0	Sample Custody
Section 8.0	Analytical Calibration Procedures and Frequency
Section 9.0	Analytical Procedures
Section 10.0	Data Reduction, Validation and Reporting
Section 11.0	Internal Quality Control Checks
Section 12.0	Performance and Systems Audits
Section 13.0	Preventive Maintenance
Section 14.0	Specific Procedures Used to Assess Data Precision, Accuracy and Completeness
Section 15.0	Corrective Action
Section 16.0	Quality Assurance Reports to Management
Appendix I	Dioxin-Furans Method Modification

LIST OF TABLES

<u>Table</u>	<u>Page</u>
5.1 Accuracy and Precision Objectives.....	18
8.1 Concentration Calibration Solutions.....	28
8.2 Criteria for Isotopic Ratio Measurements for PCDDs/PCDFs.....	29
12.1 Summary of Performance and System Audits.....	46

LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
4-1 Enseco Incorporated Quality Assurance Organizational Chart.....	9
4-2 Project Organization Chart.....	10
4-3 Project Flow.....	11
7-1 Enseco Sample Processing Flow Chart.....	23
7-2 Chain-of-Custody Record.....	24
10-1 Data Validation Scheme.....	38
11-1 Laboratory Performance Quality Control Sample Evaluation.....	42

### 3.0 PROJECT DESCRIPTION

The United States Environmental Protection Agency (US EPA) has issued an Administrative Order of Consent (Order) to CIBA-GEIGY Corporation pursuant to the Resource Conservation and Recovery Act (RCRA). The Order (No. I-88-1088) requires that a RCRA Facility Investigation be conducted at the CIBA-GEIGY facility in Cranston, Rhode Island. The order was signed by Ciba-Geigy Corporation on 9 June 1989 and became effective on 16 June 1989.

The RCRA Facility Investigation (Facility Investigation) is one phase of the RCRA corrective action program. That program also consists of the RCRA Facility Assessment (Facility Assessment) which precedes the Facility Investigation and the Corrective Measures Study (CMS) which follows the Facility Investigation.

The Facility Assessment is intended to identify and gather information on known or potential release, evaluate Solid Waste Management Units (SWMUs) and Areas of Concern, and make preliminary determinations regarding conditions of concern and the need for further action including interim measures. Those measures are designed to mitigate potential or actual releases that could endanger human health and/or the environment.

The Facility Investigation is conducted to characterize the impact of known or suspected releases that were determined to require further action based on the Facility Assessment. The Facility Investigation includes the Risk Evaluation. The Risk Evaluation is designed to identify the human populations and environmental systems that may be impacted by conditions of concern associated with the facility.

The Media Protection standards are then established for each media of concern. The Media Protection standards are based on the Risk Evaluation, promulgated standards and non-promulgated criteria.

The Corrective Measures Study determines the potential engineering solutions to the facility problems as indicated by the Media Protection Standards. The solutions (corrective measures) are evaluated based on performance, reliability, ease of implementation, timeliness, protection of human health and the environment and cost effectiveness.

Radian Corporation has been contracted by Ciba-Geigy to perform analysis of field samples for this Ciba-Geigy RCRA Facility Investigation. Radian will also furnish sample bottles, chain-of-custody forms, chain-of-custody seals, sample labels, preservation reagents, blue ice, coolers, and Radian laboratory return labels for return shipment of samples.

This analytical Quality Assurance Project Plan (QAPP) addresses analyzing a subset of the project samples for tetra through octa dibenzo-p-dioxins and dibenzofurans analyzed by Enseco-Cal Lab for verification of Radian's results. The remaining sample after Radian's analysis from 7 field locations obtained during Round 1 were sent to Enseco-Cal Lab. Split samples were taken during Round 2 and concurrently sent to Radian and Cal.

The purpose of this Quality Assurance Project Plan (QAPP) is to describe the methods and procedures that were used by Enseco-Cal Lab to ensure quality, precision, accuracy, and completeness of the analytical data generated as part of the RCRA Facility Investigation. The analytical data which will be generated by Enseco-Cal Lab will come from chemical analysis of water, soil, and sediment samples collected during Phase IA and IB of the Facility Investigation. Details regarding the scope and intent of the Facility Investigation are contained in the Facility Investigation Work Plan (Volume 1, Chapter 3, Section 4). During Phase IB samples will be collected and analyzed for chemical and treatability parameters. The sources of these samples and their associated analyses are summarized in Tables 4.2 through 4.5 of the Facility Investigation Work Plan.

This QAPP is based on the USEPA Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80 and also reflects the provisions of the CIBA-GEIGY Corporation Project Quality Assurance Plan.

This QAPP focuses on the acquisition of environmental data of defined and acceptable quality.

#### 4.0 RESPONSIBILITIES AND AUTHORITIES

Project organization is shown in Figure 4-2. The special analytical services group hierarchy is shown in Figure 4-1 and a schematic of the project work flow is shown in Figure 4-3. Responsibilities are outlined as follows:

##### DIVISIONAL QUALITY ASSURANCE DEPARTMENTS

###### Members

Each Divisional QA Department is managed by a QA Director. The QA Director reports directly to the Regional General Manager and indirectly to the Corporate QA Director. The QA Director is supported by a QA staff within the laboratory. The Regional General Manager is the final authority within each laboratory on all issues dealing with data quality. He/she has the authority to require that procedures be amended or discontinued or analyses suspended or repeated. He/she can make recommendations to the Regional General Manager and the Corporate QA Director regarding suspension or termination of employees for incompetence or non-compliance with QA procedures. The authority of the Division QA Director comes directly from the Corporate QA Director.

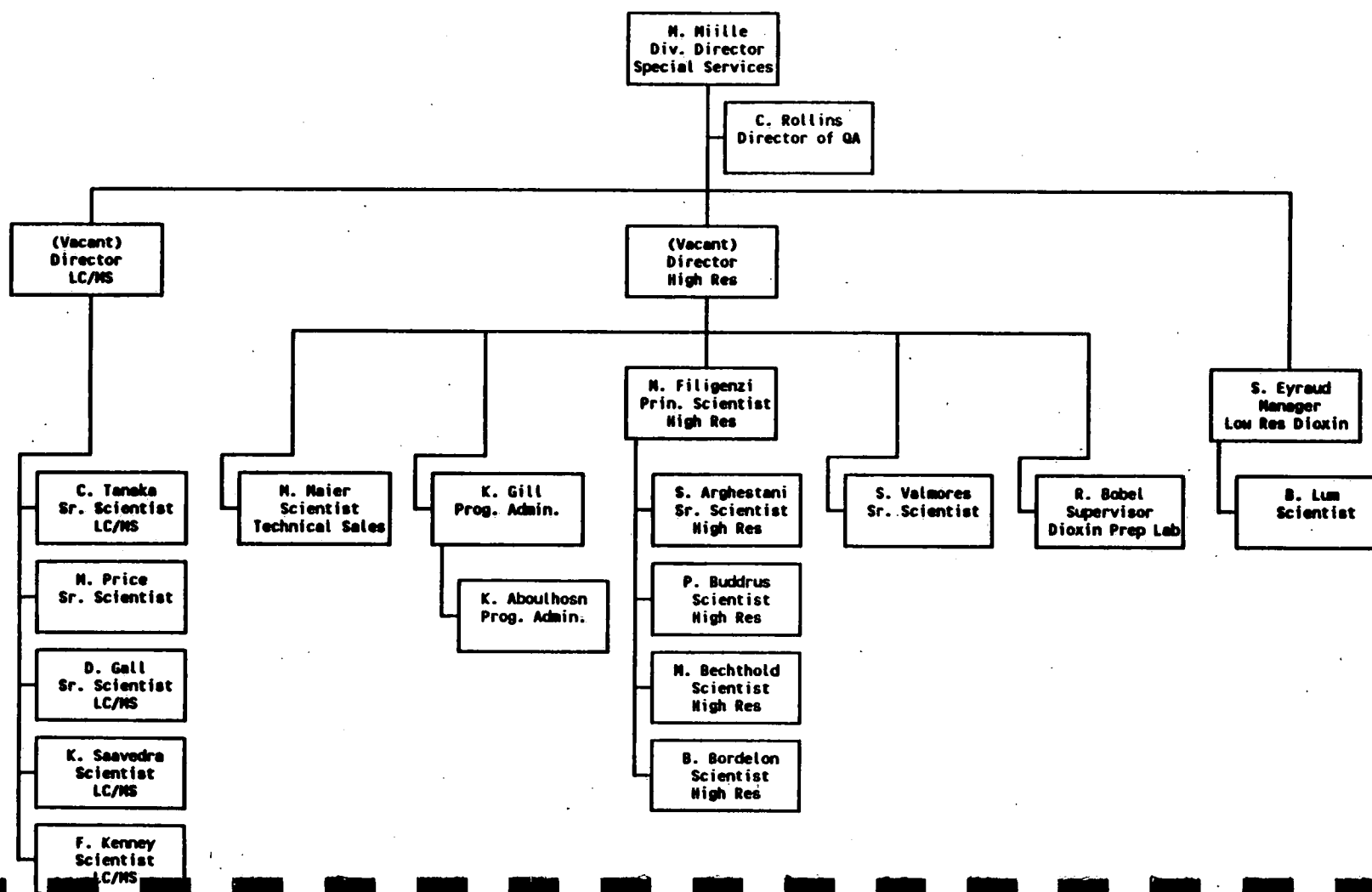
##### DIVISIONAL MANAGEMENT

###### Members

The managers, supervisors, department directors, and program administrators who direct the analytical work at each laboratory are directly responsible for ensuring that all employees reporting to them are complying with the Enseco QA Plan.

FIGURE 4-1

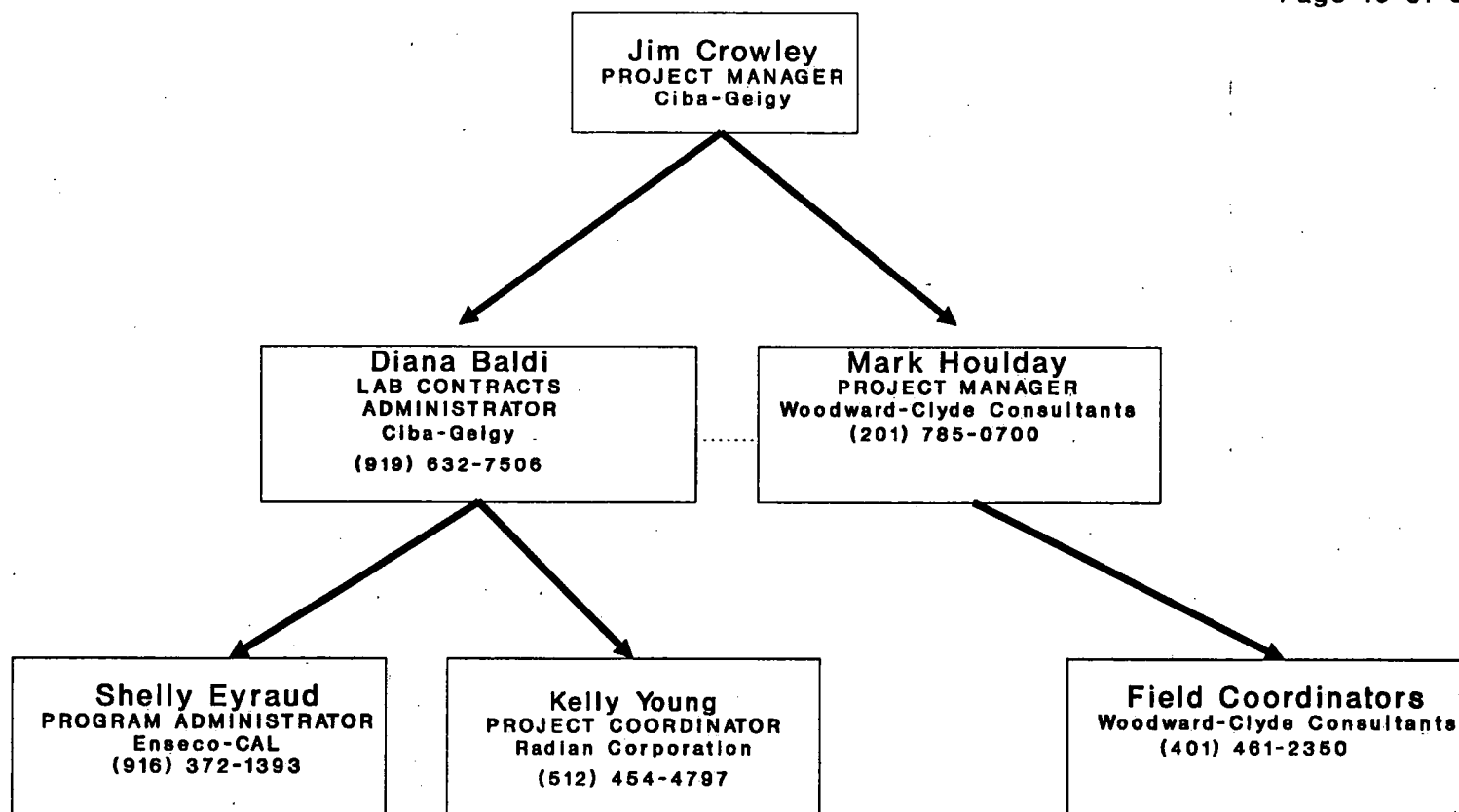
ENSECO INCORPORATED QUALITY ASSURANCE ORGANIZATIONAL CHART



# Project Organization

Figure 4.2

Section 4.0  
Revision 1  
May 1991  
Page 10 of 56

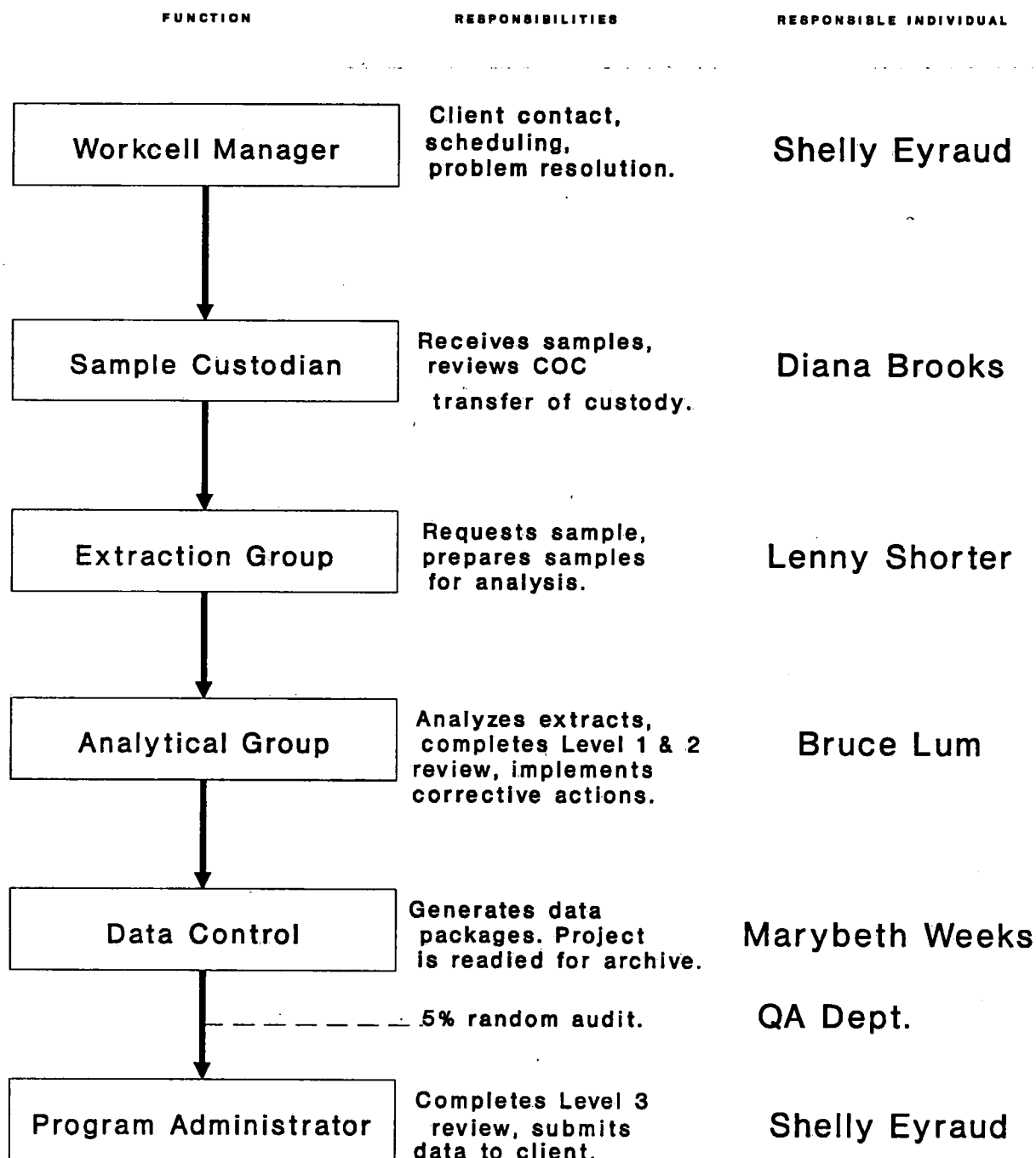


# Project Flow

## Figure 4.3

Section 4.0  
Revision 0  
May 1991  
Page 11 of 56

### Dioxin/Furans Workcell



The managers and supervisors of the laboratory have the authority to accept or reject data based on compliance with well-defined QC criteria. In addition, managers and supervisors, with the approval of the QA department, can accept or reject data that fall outside of established QC guidelines if, in their judgement, there are technical reasons which warrant the acceptance or rejection of the data. These circumstances must be well documented and any need for corrective action identified by the incident must be defined and initiated. The authority of the laboratory management comes directly from the President and the Regional General Manager.

#### DIVISIONAL PERSONNEL

##### Members

All laboratory personnel (including chemists, managers, etc.) involved in the generation and reporting of data have a responsibility to understand and follow the Enseco QA Plan.

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC criteria. The acceptance or rejection of data that fall outside of established QC guidelines must be approved by laboratory management and the QA department. The authority of the laboratory personnel flows from the Regional General Manager.

5.0 QUALITY ASSURANCE OBJECTIVES  
FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS,  
REPRESENTATIVENESS AND COMPARABILITY

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. These terms are described as follows:

DCS = Duplicate control samples, a pair of standard, control matrix that is spiked with a group of target compounds.

RPD = Relative percent difference.

RSD = Relative standard deviation.

CV = Coefficient of variation.

S = Standard deviation.

X = A measured value.

$\bar{X}$  = Average. Calculated as the sum of all measured values in a population divided by the number of values in the population.

n = Number of measurements or values in a population.

Precision is the degree to which the measurement is reproducible.

Precision can be assessed by replicate measurements of DCS, reference materials, or environmental samples. Enseco routinely monitors precision by comparing the RPD between DCS measurements with control limits established at plus three standard deviations from the mean RPD of historical DCS data.

Precision is frequently determined by comparison of replicates. The standard deviation of "n" measurements of "x" is commonly used to estimate precision.

Standard deviation (S) is calculated as follows:

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^N (X_i - \bar{X})^2}$$

where a quantity "x" (e.g., a concentration) is measured "n" times.

The relative standard deviation (or sample coefficient of variation, CV), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of  $n = 2$ ).

$$RSD = 100 (s/\bar{X})$$

or

$$CV = 100 (s/\bar{X})$$

where: RSD = relative standard deviation

CV = coefficient of variation

s = Standard deviation

$\bar{X}$  = mean

In the case of duplicates, the RPD between the two samples may be used to estimate precision.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference

D<sub>1</sub> = first sample value

D<sub>2</sub> = second sample value (duplicate)

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using DCS, standard reference materials, or spiked environmental samples. Unless specified otherwise in special contracts, Enseco monitors accuracy by comparing DCS results with control limits established at plus or minus three standard deviation units from the mean of historical LCS results.

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} = \frac{X}{T} \times 100$$

where: X = the observed value of measurement

T = "true" value

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Enseco strives to accommodate all sample matrices. Some samples may require analysis of multiple phases to obtain representative results.

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.

To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the data base is sufficient.

When possible, the percent completeness for each set of samples is calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100\%$$

The completeness objective is 100%. Reanalysis will be performed in accordance with the procedures stated in Section 9.0, Analytical Procedures, and in Appendix I in order to meet this completeness goal.

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), consistency in reporting units (ppm, ppb, etc.), and analysis of standard reference materials.

For this project Method 8280 was requested. See Appendix I for the list of enhancements to Method 8280 that are part of Enseco-Cal Lab's procedure.

Specific Data Quality Objectives are addressed in the Project QA Plan, Volume 3 of this project document. Method specific accuracy and precision objectives are listed in Table 5.1.

TABLE 5.1  
ACCURACY AND PRECISION OBJECTIVES

<u>COMPOUND</u>	<u>SPIKE CONCENTRATION (ng)</u>	<u>% RECOVERY</u>	<u>RPD</u>
2,3,7,8-TCDF	10	60-140	50
1,2,3,7,8-PeCDF	10	60-140	50
1,2,3,4,7,8-HxCDF	10	60-140	50
1,2,3,4,6,7,8-HpCDF	10	60-140	50
OCDF	50	60-140	50
2,3,7,8-TCDD	10	60-140	50
1,2,3,7,8-PeCDD	10	60-140	50
1,2,3,4,7,8-HxCDD	10	60-140	50
1,2,3,4,6,7,8-HpCDD	10	60-140	50
OCDD	50	60-140	50

Units = ng/sample

## 6.0 SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, and therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

- Samples must be collected in appropriate containers. In general, glass containers are used for organic parameters;
- The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;
- Samples must be preserved appropriately to minimize the loss of materials of interest due to adsorption, chemical or biological degradation, or volatilization;
- Appropriate volumes of sample must be collected to ensure that the required detection limits can be met and quality control samples can be analyzed;
- Samples must be properly shipped to the laboratory, in the appropriate time frame, to ensure that holding times for the analyses can be met.

Specific site sampling procedures are addressed in the Project QA Plan, Volume 3 of this project document.

### Sample Containers and Preservatives

Enseco can assist in the sample collection process by providing consultation and assistance to client designing sampling programs. Also, Enseco can make available to the client the Enseco "Sample Safe™", a set of sample containers that are properly cleaned and preserved for use in sample collection.

EPA has established holding time requirements for some analyses. However, holding times for dioxins/furans can vary depending upon the method requested. EPA Method 1613A states that up to one year before extraction of aqueous and solid samples is acceptable due to the stability of dioxins and furans in the environment. This is consistent with the scientific literature which contains several references to the half-life of dioxins in soil - all of which measure the time in years. Samples will be extracted and analyzed according to a client's contract specifications.

On occasion, a sample must be reanalyzed to comply with this QA Program Plan. If this reanalysis is conducted outside of the holding time, the laboratory will be considered to have fulfilled its obligation to meet holding times if the first preparation and/or analysis was initiated within the prescribed holding time.

## 7.0 SAMPLE CUSTODY

A sample is considered in custody if:

- It is in the sampler's or the transferee's actual possession;
- It is in the sampler's or the transferee's view, after being in his/her physical possession;
- It was in the sampler's or the transferee's physical possession and then he/she secured it to prevent tampering; and
- It is placed in a designated secure area.

Upon transfer of custody to Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

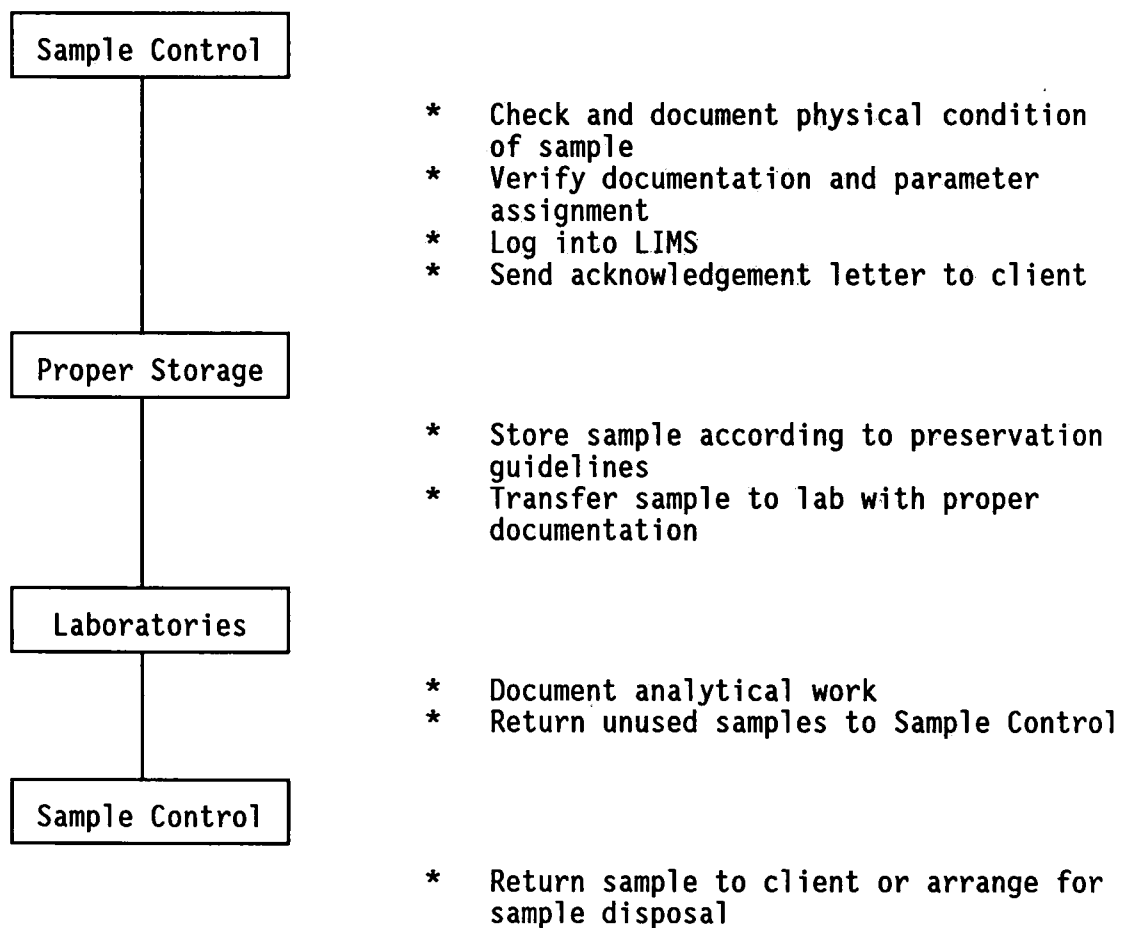
All samples are received by Enseco's Sample Control Group's designated sample custodian and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

In the event that the laboratory sample custodian judges the sample custody to be invalid (e.g., samples arrive damaged or custody seals have been broken), the Program Administrator (PA) will be advised immediately and the samples will not be analyzed until the PA so authorizes. The PA or designated representative will immediately contact the Woodward-Clyde Site Manager, Mark Houlday. The PA and the Site Manager will make a decision as to the fate of the sample(s) in question on a case-by-case basis. The sample(s) will either be processed "as is" with custody failure noted along with the analytical data, or rejected with sampling rescheduled if necessary. The Site Manager will schedule any resampling. Any problem with a sample will be noted on the chain-of-custody form.

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2.

FIGURE 7-1

ENSECO SAMPLE PROCESSING FLOW CHART



**FIGURE 7-2**  
**CHAIN-OF-CUSTODY RECORD**

**Enseco**

Page \_\_\_\_ of \_\_\_\_

**CHAIN-OF-CUSTODY RECORD**

SAMPLER: (Signature) \_\_\_\_\_

Date Shipped _____	Carrier _____
Phone _____	Cooler No. _____
Airbill No. _____	

**SHIP TO:**

 Enseco-Cal Lab  
 2544 Industrial Blvd.  
 West Sacramento, CA 95691  
 (916) 372-1393

**SEND RESULTS TO:**

 Client Name \_\_\_\_\_  
 Company \_\_\_\_\_  
 Address \_\_\_\_\_  
 Phone \_\_\_\_\_

ATTENTION: \_\_\_\_\_

PROJECT NAME _____	PROJECT NO. _____	P.O. NO. _____
--------------------	-------------------	----------------

Relinquished by: (Signature) _____	Received by: (Signature) _____	Date _____	Time _____
------------------------------------	--------------------------------	------------	------------

Relinquished by: (Signature) _____	Received by: (Signature) _____	Date _____	Time _____
------------------------------------	--------------------------------	------------	------------

Relinquished by: (Signature) _____	Received at lab by: (Signature) _____	Date _____	Time _____
------------------------------------	---------------------------------------	------------	------------

Relinquished from lab by: (Signature) _____	Received by: (Signature) _____	Date _____	Time _____
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**ANALYSIS REQUEST**

Sample ID Number	Sample Description	Date Time Sampled	Analysis Requested	Sample Condition Upon Receipt

Special Instructions Comments: \_\_\_\_\_

**NOTE: UNUSED PORTIONS OF NON-AQUEOUS SAMPLES WILL BE RETURNED TO CLIENT**

Expected Analytical T.A.T.s	Immediate Attention (200% surcharge) _____ _____ RUSH (50-100% surcharge) _____ _____ Standard _____
-----------------------------	--

Cal Lab ID Number: (for lab use only) \_\_\_\_\_

## 8.0 ANALYTICAL CALIBRATION PROCEDURES AND FREQUENCY

### Standard/Reagent Preparation

To ensure the highest purity possible, all primary reference standards and standard solutions used by Enseco are obtained from the National Institute of Standards and Technology, the EPA Repository or other reliable commercial sources. Dioxins/furans standards will be purchased from Cambridge Isotope Laboratories, Woburn, Massachusetts or from the US EPA, EMSL-Las Vegas (when available). All standards and standard solutions are logged into a data base that identifies the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory.

### Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. Specific procedures are described as follows:

### Tuning

Mass calibration of the mass spectrometer is tuned prior to the analysis of standards or samples each analysis day. The compound FC43 is used to tune the instrument for greater sensitivity in the high mass range to achieve better response in the later eluting compounds.

### Window Defining Mix (WDM)

The window defining mix is analyzed to verify that the switching times between the descriptors have been appropriately set and is analyzed for the following conditions:

- Before initial calibration on each instrument and on each gas chromatography column used for analysis.
- Each time a new initial calibration is performed, regardless of reason.
- Each time that adjustments or instrument maintenance activities are performed that may affect retention times.

### Initial Calibration

Five calibration solutions (CC1-CC5) containing 10 unlabeled and 6 carbon labeled PCDDs/PCDFs at known concentrations are used to calibrate the instrument before sample analysis can commence. Analytes and concentrations are listed on Table 8.1. The relative ion abundance ratios (using areas to calculate the ratios) must be within the limits outlined in Table 8.2. In addition, all analytes must fall within the retention time windows as determined by the WDM and meet the mass spectrometer sensitivity criteria, i.e., the signal-to-noise ratio must be greater than 2.5 for the unlabeled PCDDs/PCDFs ions and greater than 10 the the internal standard ions. The %RSD of the RRF's for the unlabeled PCDDs/PCDFs, the surrogate, and the internal standards must not exceed 15%. Formulas used to calculate %RSD and RRF are listed in Section 14.0.

Continuing Calibration Standard (Daily Standard)

The daily standard is analyzed every 12 hours to demonstrate continued acceptable GC/MS performance. The RRF of the compounds in the continuing calibration standard must be within 30% of the average RRF determined from the Initial Calibration. If this criteria cannot be met, analysis is suspended, the problem investigated, corrective actions implemented, and a new 5 point calibration is performed, if needed.

Optimum Range

The optimum concentration range of this method is 0.5 - 10 ppb.

**TABLE 8.1**
**Concentration Calibration Solutions\*  
 Used for Initial Calibration**

	Low	Mid-Low	Med.	Mid-high	High
2,3,7,8-TCDF	0.2	0.5	1.0	2.0	5.0
1,2,3,7,8-PnCDF	0.2	0.5	1.0	2.0	5.0
1,2,3,6,7,8-HxCDF	0.2	0.5	1.0	2.0	5.0
1,2,3,4,6,7,8-HpCDF	0.2	0.5	1.0	2.0	5.0
OCDF	0.40	0.50	2.0	4.0	5.0
2,3,7,8-TCDD	0.2	0.5	1.0	2.0	5.0
1,2,3,7,8-PnCDD	0.2	0.5	1.0	2.0	5.0
1,2,3,6,7,8-HxCDD	0.2	0.5	1.0	2.0	5.0
1,2,3,4,6,7,8-HpCDD	0.2	0.5	1.0	2.0	5.0
OCDD	0.40	0.50	2.0	4.0	5.0
13C-2,3,7,8-TCDF	0.5	0.5	0.5	0.5	0.5
13C-2,3,7,8-TCDD	0.5	0.5	0.5	0.5	0.5
13C-1,2,3,7,8-PnCDD	1.0	1.0	1.0	1.0	1.0
13C-1,2,3,6,7,8-HxCDD	1.0	1.0	1.0	1.0	1.0
13C-1,2,3,4,6,7,8-HpCDD	1.0	1.0	1.0	1.0	1.0
13C-OCDD	5.0	5.0	5.0	5.0	5.0
37Cl-2,3,7,8-TCDD	0.2	0.2	0.2	0.2	0.2

\* Concentrations are in ng/ul.

TABLE 8.2  
 Criteria for Isotopic Ratio Measurements  
 for PCDDs and PCDFs

	<u>Analyte</u>	<u>Selected ions</u>	<u>Relative Intensity</u>
PCDFs	Tetra	304/306	0.65-0.89
	Penta	340/342	1.31-1.77
	Hexa	374/376	1.05-1.41
	Hepta	408/410	0.88-1.18
	Octa	442/444	0.75-1.01
PCDDs	Tetra	320/322	0.65-0.89
	Penta	356/358	1.31-1.77
	Hexa	390/392	1.05-1.41
	Hepta	424/426	0.88-1.18
	Octa	458/460	0.75-1.01
Internal Standards			
	13C-TCDF	316/318	0.65-0.89
	13C-2,3,7,8-TCDD	332/334	0.65-0.89
	13C-PnCDD	368/370	1.31-1.77
	13C-HxCDD	402/404	1.05-1.41
	13C-HpCDD	436/438	0.88-1.18
	13C-OCDD	470/472	0.75-1.01
Recovery Standard			
	13C-1,2,3,4-TCDD	332/334	0.65-0.89

## 9.0 ANALYTICAL PROCEDURES

The methods used are those specified by the US EPA and other federal agencies, state agencies, and professional organizations, as provided in the following references:

- Current EPA (CLP) protocols for the analysis of organic and inorganic hazardous substances including chlorinated dioxins and furans.
- "Test Methods for Evaluating Solid Waste" (SW-846), 2nd Edition (revised), Update I (1984), Update II (1985), 3rd Edition (1986), Update I (1989), Office of Solid Waste and Emergency Response, US EPA.

The choice of method is dependent on the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, and the type of matrix to be analyzed. Each method used routinely is documented in the form of an SOP. The SOP contains detailed instructions concerning both the use and the expected performance of the method. The method selected will detect and quantify 2,3,7,8-tetrachlorinated dibenzo-p-dioxin (2,3,7,8-TCDD), 2,3,7,8-tetrachlorinated dibenzofuran (2,3,7,8-TCDF), and the 2,3,7,8-substituted penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs). Any deviations from published methodology are documented and explained in Appendix I. Samples that initially do not meet quality assurance objectives will be reanalyzed once to verify observed anomalies. Additional reanalysis with modifications can be performed at the request of the client for an appropriate fee. Specific analytical procedures are as follows:

### Internal Standards

To quantitate and measure recovery of the analytes, labeled internal standards will be added to all pre-extraction samples, QC samples, blanks, extraction, and to calibration solutions. Calculated internal standard recoveries must be  $\geq 40\%$  or the signal-to-noise ratio must be at least 10:1 before data is considered acceptable. Otherwise, samples will be reextracted and reanalyzed at a smaller sample volume. Associated analytes and internal standards are listed in Appendix I.

### Surrogate

The surrogate 37Cl-2,3,7,8-TCDD will be added to all pre-cleanup blanks, samples, QC samples, and to calibration solutions. The calculated % recovery is used to verify the recovery of unlabeled PCDDs/PCDFs and to monitor the efficiency of the cleanup procedures.

### Recovery Standard

To measure the % recovery of the labeled internal standards, the recovery standard 13C-1,2,3,4-TCDD will be added to all blank, sample, and quality control sample extracts just prior to GC/MS analysis.

### Additional Quality Control

In addition, the following quality control samples will be analyzed:

- Method blanks
- Matrix spike/matrix spike duplicate sample
- Duplicate control sample (DCS)

Definitions, frequency, and corrective action are discussed in Section 11.0, Internal QC Checks and Frequency. Acceptability criteria are listed in Table 5.1, Accuracy and Precision Objectives.

### Calculations

A summary of calculations used to determine PCDF/PCDD recoveries and concentrations are listed in Section 14.0, Specific Procedures Used to Assess Data Precision, Accuracy, and Completeness.

### Identification

For a gas chromatographic peak to be unambiguously identified as a PCDD/PCDF, it must meet the following criteria:

- The absolute retention times must be within method acceptance limits.
- All of the specified ions listed in Appendix I for each PCDD/PCDF must be present in the ion current profile. The ion current response for the two quantitation ions and the M-[COCl]<sup>+</sup> ions for the analytes must maximize simultaneously.
- The integrated ion current for each analyte ion listed in Appendix I must be at least 2.5 times background noise with no detector saturation.
- Ion abundance ratios must meet the criteria listed in Table 8.2.

### Confirmation

Because specificity for all of the isomers cannot be achieved on a 60 M DB-5 chromatographic column, a second column (SP-2331) will be used to confirm the presence of any 2,3,7,8-substituted PCDDs/PCDFs detected. Samples are first analyzed on a GC/MS fitted with a 60 M DB-5 chromatographic column. If any 2,3,7,8-substituted tetra-, penta-, or hexa- PCDDs/PCDFs are detected, the sample extracts will be reanalyzed using a 60 M SP-2331 chromatographic column. If data resulting from SP-2331 column does not confirm results from the DB-5 column, only data calculated from the SP-2331 column will be reported. All data will be corrected following proper error correction protocol.

## 10.0 DATA REDUCTION, VALIDATION, AND REPORTING

All analytical data generated within Enseco laboratories are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review, as described below (also see Figure 10-1).

### Level 1 Review

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs.

Data will be reduced by an analyst in one of the following ways:

- Manually computing results directly on the data sheet, chromatogram, or on calculation pages attached to the raw data;
- Inputting raw data for computer processing.

If data are manually reduced by an analyst, all steps in the computation will be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and RFs, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than).

Finally, each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample identification is correct;
- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met;
- Congener identification is correct; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, Out-of-Control forms [if required] are complete; holding times are documented, etc.).

The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated Level 1 review. The analyst then passes the data package to an independent reviewer, who performs a Level 2 review.

### Level 2 Review

Level 2 review is performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; Out-of-Control forms [if required] are complete; holding times are documented, etc.).
- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Level 2 review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

### Level 3 Review

Before the report is released to the client, the Program Administrator who is responsible for interfacing directly with the client reviews the report to ensure that the data meet the overall objectives of the client, as understood by the Program Administrator. This review is labeled Level 3 review.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgement of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

In addition to the three levels of review discussed above, the Divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. Errors that are found are documented and transmitted to the appropriate supervisors and managers. The cause of the errors is then addressed with additional training or clarification of procedures to that quality data is generated from the lab. The process continues until no errors are found or until the data package has been reviewed in its entirety.

### Detection Limits

The detection limit will be calculated for the 10 congeners when no unlabeled PCDFs/PCDDs are detected in the samples. The process is discussed in Appendix I. Calculations are listed in Section 14.0.

## Reporting

Results will be reported to the second decimal place in units of ng/l (ppt) for aqueous samples, and ng/g (ppb) for solid samples on a wet weight basis.

An example of a dioxin/furan data validation scheme is shown as Figure 10-1.

## Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. In general, Enseco reports contain:

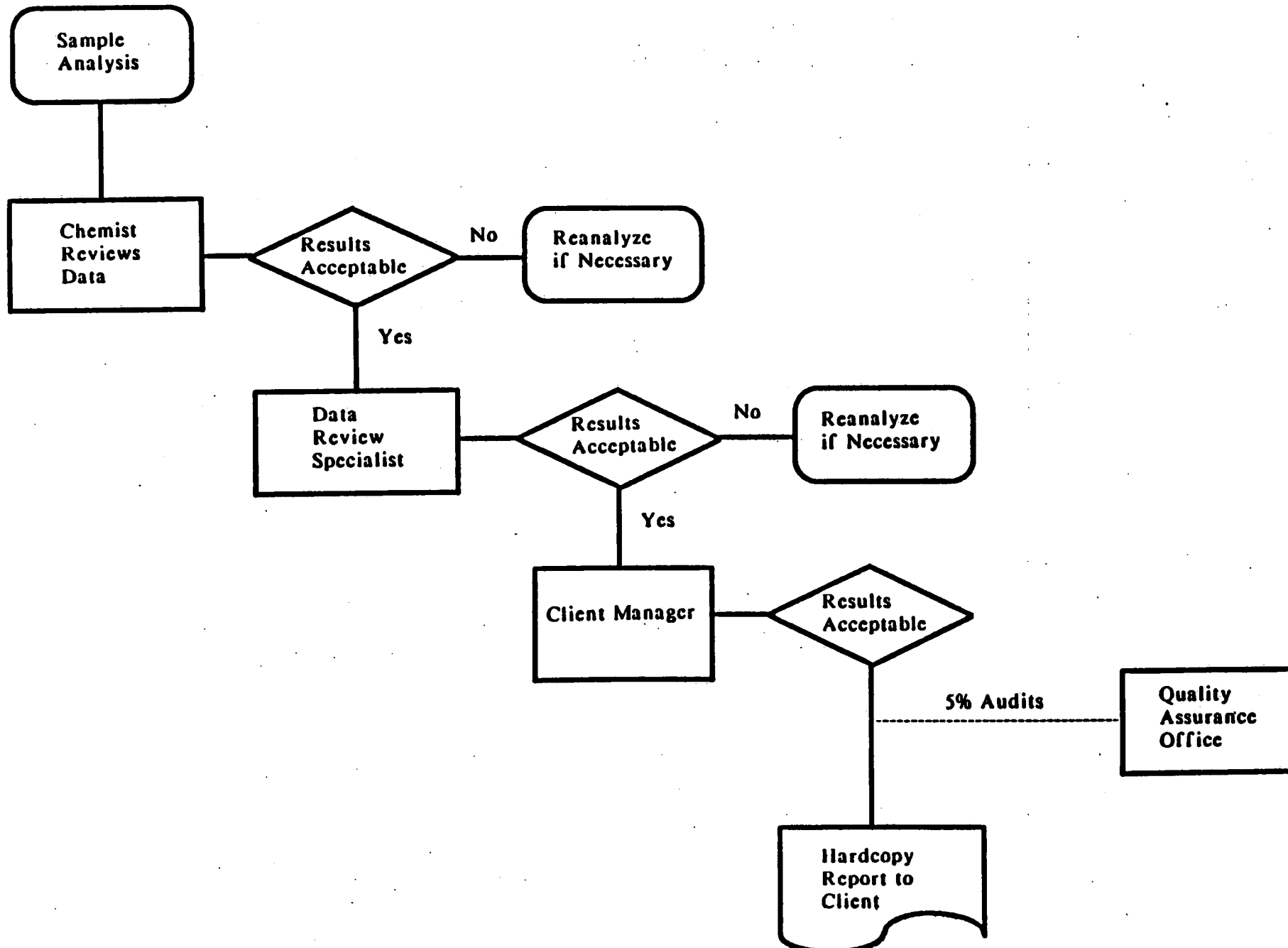
- General Discussion: Description of samples types, tests performed, any problems encountered and general comments are given.
- Analytical Data: Data are reported by sample or by test. Pertinent information including dates sampled, received, prepared, and extracted are included on each results page. The Enseco reporting limit for each analyte is also given.
- QC Information: The results (Percent Recovery and Relative Percent Difference) of the Laboratory Control Samples analyzed with the project are listed, together with the control limits. Also, the analytical results for method blanks generated during analysis of organic and metals parameters are given.

Results of any matrix spikes, duplicates, matrix spike duplicates or other project-specific QC are also reported.

- Methodology: Reference for analytical methodology used is cited.
- Raw Data: Including calibration data, window defining mix data are included in CLP-type deliverables.

Figure 10-1

## Data Validation Scheme



### Subcontractor Validation

After undergoing the laboratories internal data validation, reduction, and reporting process, staff chemists at Woodward Clyde Consultants will validate the data using "Organics Analytical Data Review, US EPA Region 1 Worksheets Edited for Appendix 9 Compounds, Prepared by Woodward Clyde Consultants, 04 February 1991".

### 11.0 INTERNAL QC CHECKS

Laboratory Performance QC samples will be added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration so that accuracy and precision can be determined and control charts can be prepared. Measures taken to control analytical data quality include use of specific acceptance criteria for instrument calibration, laboratory control samples, duplicate analyses, blank samples, and spiked samples.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. The main elements of Laboratory Performance QC are:

- The analysis of Laboratory Control Samples, which include Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks; and
- The generation of daily calibration data.

Duplicate Control Samples (DCS) are used to monitor the precision and accuracy of the analytical system on an on-going basis. Each DCS consists of a standard, control matrix that is spiked with the internal standards and the surrogate representative of the method analytes. A DCS pair is analyzed for every 20 samples processed by the method. DCS are analyzed with environmental samples to provide evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

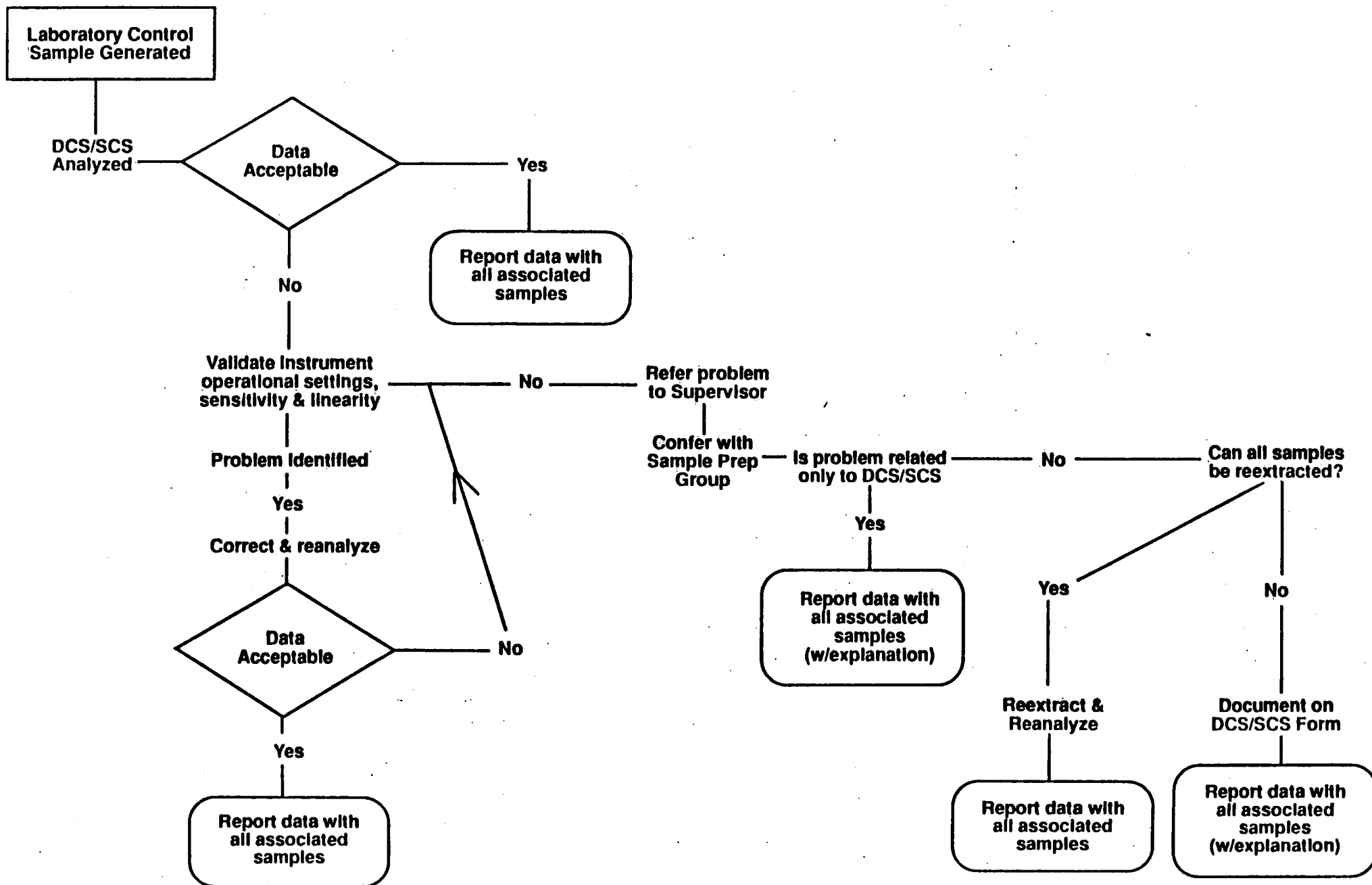
Accuracy (average recovery of each analyte in the DCS pair) and precision (Relative Percent Difference [RPD] between each analyte in the DCS pair) data are compared to control limits that have been established for each of the analytes contained in the DCS. Initially, control limits for analytes spiked into the DCS are taken directly from the CLP program. If CLP limits are not available, Enseco historical data are used to set the control limits. As sufficient laboratory data become available, the control limits are redefined based upon the most recent nine months of DCS data. Control limits for accuracy for each analyte are based on the historical average recovery (mean of the average recoveries of the DCS pairs) plus or minus three standard deviation units. Control limits for precision for each analyte are based on the historical RPD and range from zero (no difference between DCS results) to the average RPD plus three standard deviation units.

Analytical data that are generated with a DCS pair which falls within the established control limits are judged to be in control. The procedure used to evaluate data from control samples is given in Figure 11-1. The protocols include examination of instrument performance and preparation and analysis information, consultation with the supervisor, and finally a decision path for determining whether reanalysis is warranted.

#### Method Blank

Method blanks, also known as reagent, analytical, or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

# Laboratory Performance QC Control Sample Evaluation



As part of the standard Enseco QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, cleanup, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

A method blank is prepared and analyzed with every analytical lot or for every 20 samples, whichever is more frequent. Any analyte detected in the blank must be below the reporting limit, otherwise samples are reextracted and reanalyze.

#### Matrix-Specific QC

Matrix-Specific QC is used to assess the effects of a sample matrix or field conditions on the analytical data.

Different regulatory programs have different requirements in terms of Matrix-Specific QC. In order to ensure that the data generated meet all Data Quality Objectives, Enseco encourages its clients to include Matrix-Specific QC that fulfills the Data Quality Objectives and regulatory requirements of the project. A discussion of the different elements of Matrix-Specific QC follows.

#### Matrix Spikes and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

A Matrix Spike Duplicate (MSD) is an environmental sample that is divided into two separate aliquots, each of which is spiked with known concentrations of the analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

#### Surrogate Compounds

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. A labeled surrogate will be added to all extracted, pre-cleanup blanks, samples, QC samples, and to calibration solutions to monitor the efficiency of the cleanup procedure.

#### Field Blank

Field blanks are check samples that monitor contamination originating from the collection, transport or storage of environmental samples. Solvents such as trichloroethylene are the medium of choice for field blanks when sampling for dioxins and furans; however, if solvents are also parameters of interest, no field blank will be collected. One example of a field blank is an equipment blank. Another type of field blank is a trip blank. A trip blank is a laboratory control matrix (typically water) which is sent to the field in an appropriate sample container, remains unopened in the field, and then is sent back to the laboratory. The purpose of the trip blank is to assess the impact of field and shipping conditions on the samples. The results from field blanks are reported to the client as samples in the same concentration units as the samples. No correction of the analytical data is done in the laboratory based on the analysis of field blanks. The purpose of the trip blank is to assess the impact of field and shipping condition on the samples.

## 12.0 PERFORMANCE AND SYSTEMS AUDIT

Enseco laboratories participate in a variety of federal and state certification programs, (including the US EPA CLP), that subject each of the laboratories to stringent systems and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Enseco is certified to perform environmental analyses under programs administered by the US EPA, US Army, US Navy, and over 4 states. The most current list of Enseco certification is available upon request.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required.

Ciba-Geigy, Inc. performed both a system and a performance audit for this project. Two 2,3,7,8-TCDD performance evaluation samples were supplied by EPA Region I and submitted to the laboratory for concurrent analysis with samples. Diana Baldi and Frank Saksa of Ciba-Geigy conducted a systems audit while samples were being processed by the laboratory.

A summary of the types and frequency of systems and performance audits is summarized in Table 12.1.

TABLE 12.1

## SUMMARY OF PERFORMANCE AND SYSTEM AUDITS

<u>PROGRAM</u>	<u>NAME</u>	<u>TYPE (P OR S)</u>	<u>FREQUENCY</u>
Drinking water	WS	Performance	Semi-Annual
Waste water	WP	Performance	Semi-Annual
EPA-CLP	QB	Performance	Quarterly*
U.S. Navy	PE	Performance	Every 18 mos.*
U.S. Army	PE	Performance	Every 18 mos.*
Calif. Dept. Food and Agriculture	PE	Performance	Quarterly
NPDES	DMR-QA	Performance	Annually
EPA-CLP		System	Annually*
Calif. ELAP		System	Biannually
U.S. Navy		System	Annually
Utah		System	Annually
U.S. Army		System	Every 18 mos.*
Divisional QA		System	Quarterly (approx.)
ENSECO Corporate		System	Annually
Ciba-Geigy, Inc.		System	Random

Clients may request performance of specific performance and systems audits as a requirement of contract award.

\* Contract award required.

### 13.0 PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument.

Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the instrument manufacturer.

Each laboratory has detailed SOPs on file that describe preventive maintenance procedures and schedules.

All aspects of routine and non-routine instrument maintenance are recorded in logbooks, and a log book is dedicated to each instrument.

Balances are calibrated daily or as used with Class S or Class S traceable weights at specific weights of use with the results entered in a logbook kept near the balance.

Ovens and refrigerators are fitted with uniquely marked thermometers and monitored daily. Limits for refrigerators are 2°C to 6°C. If a temperature falls outside these limits, the appropriate laboratory manager is alerted and corrective action is taken. The readings are entered in a logbook kept near the thermometer. Annually the thermometer is calibrated vs. an NIST traceable thermometer.

## 14.0 SPECIFIC PROCEDURES USED TO ASSESS DATA PRECISION

### ACCURACY, AND COMPLETENESS

Calculations for accuracy (% R) and precision (RPD, RSD) were discussed and defined in Section 5.0 Data Quality Objectives. Formulas used to calculate relative response factors (RRF), concentration, and detection limits are as follows:

#### Definitions

Cn = concentration of native PCDD/PCDF found in the sample

Cis = concentration of internal standards

W = weight (g) of sample extracted

V = volume (mL) of sample extracted

Qis = quantity (ng) of internal standard added to sample  
before extraction

Qrs = quantity (ng) of recovery standard added to sample extract

Qs = quantity (ng) of surrogate added to sample before extraction

An = integrated ion abundance of the quantitation ion of the  
isomer of interest (Table 1).

Ais = integrated ion abundance of the quantitation ion of the  
appropriate internal standard (Table 1 and 2).

RRFn is the response factor of the quantitation ion of the isomer  
of interest relative to that of the appropriate internal standard.

RRFs is the response factor of the quantitation ion of the  
surrogate relative to that of the appropriate internal standard.

RRFis is the response factor of the internal standard relative to  
that of the appropriate recovery standard.

### Relative Response Factors

$$RRF_n = \frac{A_n \times C_{is}}{A_{is} \times C_n}$$

$$RRF_{is} = \frac{A_{is} \times C_{rs}}{A_{rs} \times C_{is}}$$

$$RRF_s = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

### Concentration

The concentrations of the various isomers of each congener shall be calculated using the RRF determined for that particular isomer from the initial calibration.

$C_n \text{ (ng/g)} = \frac{Q_{is} \times A_n}{W \times A_{is} \times RRF_n}$	$C_n \text{ (ng/L)} = \frac{Q_{is} \times A_n}{V \times A_{is} \times RRF_n}$
---	---

### Detection Limits

For samples in which no unlabeled PCDFs/PCDDs are detected, calculate the detection limit for each of the 10 congeners. The DL is the concentration of a given PCDF/PCDD congener that would produce a signal with peak height of 2.5 times the background signal level, or when no interference is present, the equivalent concentration that the peak would represent if it were a PCDF/PCDD congener. The data must be carefully examined to determine which DL calculation will be used. Follow the rules below.

When only electronic noise is present, or if only one chemical peak is present at either the primary or secondary ion, use the following calculation:

$$DL = \frac{ht \text{ of the line (i.e., } 2.5 \times \text{noise}) \times Q_{is}}{H_{is} \times RRF_n \times W \text{ (or } V)}$$

When chemical peaks are present at both the primary and secondary ions that do not meet ratio criteria use formula 2 below:

$$MPC = \frac{H_x \times Q_{is}}{H_{is} \times RRF_n \times (W \text{ or } V)}$$

### 15.0 CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks, DCS or SCS contain contaminants above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department. Corrective action documentation is routinely reviewed by the of QA.

## 16.0 QA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Divisional QA Directors submit extensive monthly reports to the Corporate QA Director and the Regional General Manager. These reports include:

- The results of internal systems audits including any corrective actions taken;
- Performance evaluation scores and commentaries;
- Results of site visits and audits by regulatory agencies and clients;
- Performance on major contracts, (including CLP);
- Problems encountered and corrective actions taken;
- Holding time violations;
- Comments and recommendations; and
- A summary of the 5% QA data audits conducted.

The Corporate Director of QA submits regularly reports on the status of the QA program to the President and Regional General Manager. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.

## APPENDIX I PCDDS/PCDFS

Enseco-Cal Lab performs United States Environmental Protection Agency (US EPA) Method 8280 for polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) with some minor modifications to the published method. Since the method was first published in the Federal Register in September 1986, this method has undergone several updates and changes by EPA to keep pace with technology and method improvements during that time period.

Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60 M DB-5 column. In order to determine the concentrations of the individual 2,3,7,8-substituted isomers, the sample extract shall be reanalyzed on a 60 M SP-2331 GC column. The chromatographic resolution is evaluated using a commercially available column performance mixture containing the TCDD isomers that elute most closely with 2,3,7,8-TCDD.

### GENERAL DIFFERENCES

- Calculation of Detection Limits

The sensitivity of this method is dependent upon the level of interferences within the sample matrix. All PCDD and PCDF analyses performed for EPA since 1982 has used a technique for calculating the detection limit for each of the chlorination levels and each congener by using the noise level present in the elution window and the height of the chromatographic peak of the internal standard. Both the signal to noise and peak height are determined by the data system of the GC/MS. The result of the calculation is a detection limit that is specific to the homologous series and sample. We are not aware of any laboratories in the dioxin field that use or have used the MDL study referenced in the original 8280 method.

- Internal Standards

Due to the increased availability of the  $^{13}\text{C}$ -labeled PCDD and PCDF internal standards, Enseco-Cal Lab currently uses six instead of only the two internal standards specified and one recommended in the method. This clearly improves the quality of the data.

Internal Standards and Corresponding Analytes

$^{13}\text{C}$ -TCDD	$^{13}\text{C}$ -TCDF	$^{13}\text{C}$ -PnCDD	$^{13}\text{C}$ -HxCDD	$^{13}\text{C}$ -HpCDD	$^{13}\text{C}$ -OCDD
TCDD	TCDF	PnCDF	HxCDF	HpCDF	OCDF
37Cl-TCDD	----	PnCDD	HxCDD	HpCDD	OCDD

- Reporting Format

The reporting forms at the end of Method 8280 have been revised a number of times to improve the initial version. Enseco-Cal Lab results, while not reported on the forms in Method 8280, are in a "CLP-like" format.

- Initial Calibration

The initial calibration is performed with single injections of a five point curve.

- Acceptance of Internal Standard Recoveries

Method 8280 specifies internal standard recoveries must be 40% or greater. If recoveries do fall below 40%, then the signal-to-noise ratio is calculated. The recoveries of the internal standards are judged acceptable if the signal-to-noise ratio is greater than 10:1.

- Key Ions Used in the Analysis

Below are the ions used in the determination of PCDDs and PCDFs, they will differ from those listed in Method 8280. The quality of data will not be effected as the ions are consistently used in both the analytical standards and samples.

### Ions Specified for Selected Ion Monitoring for PCDDs and PCDFs

<u>Analyte</u>	<u>Quantitation ion</u>	<u>Confirmation ion</u>	<u>M-[COC1] +</u>
<b>PCDFs</b>			
Tetra	306	304	243
Penta	340	342	277
Hexa	376	374	311
Hepta	410	408	345
Octa	444	442	379
<b>PCDDs</b>			
Tetra	322	320	259/257
Penta	358	356	293
Hexa	392	390	327
Hepta	426	424	361
Octa	460	458	395
<b>Internal Standards</b>			
13C-TCDF	318	316	
13C-2378-TCDD	334	332	
13C-PnCDD	370	368	
13C-HxCDD	404	402	
13C-HpCDD	438	436	
13C-OCDD	472	470	
• <u>PCDD/PCDF isomers in the window defining mix for a 60 M DB-5 (or equivalent) column.</u>			
<u>Homologue</u>	<u>First Eluted</u>	<u>Last Eluted</u>	
TCDD	1368-	1238-	
TCDF	1368-	1289-	
PeCDD	12478	12389-	
PeCDF	13468-	12389-	
HxCDD	124679-	123467-	
HxCDF	123468-	123489-	
HpCDD	1234679-	1234678-	
HPCDF	1234678-	1234789-	

**SPECIFIC DIFFERENCES**• Analytical

Section 4.3.2 - 8280: 30 M DB-5

Cal: 60 M DB-5

Section 10.3 - 8280: Recommended that the GC/MS run be divided into five selected ion monitoring sections.

Cal: GC/MS run is divided into three selected ion monitoring sections.

Section 11.1 - 8280: Response factor of the quantitation ion (m/z 334) of the internal standard,  $^{13}\text{C}$ -2,3,7,8-TCDD.Cal: Response factor of the quantitation ion of the compound of interest relative to the appropriate  $^{13}\text{C}_{12}$ -labelled internal standard.• Sample Preparation

Section 9.2.5 - 8280: Soil extraction with 20 mL methanol in and 80 mL petroleum ether.

Cal: 20 mL methanol and 150 mL hexane. Acceptable internal standard recoveries have been demonstrated using hexane as an extraction solvent.

Section 9.2.5.1 - 8280: Kuderna-Danish concentration.

Cal: Concentration performed by rotary evaporation.

Section 9.3 - 9.6 8280: Regents used 20% KOH, 5% NaCl

Cal: Regents used 10N NaOH, DI H<sub>2</sub>O

The acid/base wash is performed on an optional basis. A silica/alumina column cleanup is the first column cleanup performed and follows (Section 11.3) from SOW 9/86, Rev. 8/87, Form IFB Series WA 86-K357. The second column is the carbon column cleanup. This cleanup is also from SOW 9/86, Rev. 8/87, Section 11.4.3 - 11.4.5 with omitting 5% benzene as the only modification.

Section 9.7 8280: Gravity column with 4 g of Woelm super 1 neutral alumina.

Cal: Two columns in series. The first consisting of 1 cm Na<sub>2</sub>SO<sub>4</sub>, silica gel, 4 g 44% H<sub>2</sub>SO<sub>4</sub> (silica gel, 1 g silica gel, 2 g 33% 1M NaOH/silica gel, 1 g silica gel and glasswool. The second consisting of 1 cm Na<sub>2</sub>SO<sub>4</sub>, 6g acid alumina and glasswool. The above column packing materials can be found in Section 11.3, SOW 9/86, Rev. 8/87, Form IFB Series WA 86-K357. This cleanup is mandatory.

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F.B.

